Environmental Technology Verification Protocol

HVLP Coating Equipment Generic Testing and Quality Assurance Protocol

Revision No. 1

Prepared by National Defense Center for Environmental Excellence Operated by Concurrent Technologies Corporation

for the U.S. Environmental Protection Agency

Under Contract No. DAAE30-98-C-1050 with the U.S. Army (TACOM-ARDEC) via EPA Interagency Agreement No. DW97936814



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Prepared by

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Under Contract No. DAAE30-98-C-1050 (Task 208, SOW Task 4) with the U.S. Army (TACOM-ARDEC)

via EPA Interagency Agreement No. DW97936814

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SI to English Conversions

<u>SI Unit</u>	English Unit	Multiply SI by factor to obtain English
°C	°F	(1.80 E + 01), then add 32
L	gal. (U.S.)	2.642 E - 01
m	ft	3.281 E + 00
kg	lbm	2.205 E + 00
kPa	psi	1.4504 E - 01
cm	in.	3.937 E-01
mm	mil (1 mil = 1/1000 in.)	3.937 E + 01
m/s	ft/min	1.969 E + 02
kg/L	lbm/gal. (U.S.)	8.345 E + 00

List of Abbreviations and Acronyms

ACS	American Chemical Society
ANSI	American National Standards Institute
AOAC	Association of Official Analytical Chemists
ASQC	American Society for Quality Control
ASTM	American Society for Testing and Materials
CAS	conventional air spray
CBD	Commerce Business Daily
CCEP	Coatings and Coating Equipment Program
CTC	Concurrent Technologies Corporation
DFT	dry film thickness
DI	deionized
DOI	distinctness-of-image
EPA	Environmental Protection Agency
ETF	Environmental Technology Facility
ETV	Environmental Technology Verification
HAP	hazardous air pollutant
HVLP	high-volume, low-pressure
NDCEE	National Defense Center for Environmental Excellence
NIST	National Institute for Standards and Technology
PLC	programmable logic controller
QA/QC	quality assurance/quality control
QMP	Quality Management Plan
RFT	request for technologies
SAE	Society of Automotive Engineers
SOP	standard operating procedure
TE	transfer efficiency
TQAPP	Testing and Quality Assurance Project Plan
VOC	volatile organic compound
WBS	work breakdown structure

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1.0 INTRODUCTION

1.1 Purpose of the Generic Testing and Quality Assurance Protocol

The primary purpose of this document is to establish the generic protocol for high- volume, low-pressure (HVLP) coating equipment. The secondary purpose is to establish the generic format and guidelines for HVLP Coating Equipment Testing and Quality Assurance Project Plans (TQAPPs).

Environmental Technology Verification Coatings and Coating Equipment Program (ETV CCEP) project level TQAPPs will establish the specific data quality requirements for all technical parties involved in each project. A defined format, as described below, is to be used for all ETV CCEP HVLP equipment TQAPPs to facilitate independent reviews of Project Plans and test results, and to provide a standard platform of understanding for stakeholders and participants.

1.2 Quality Assurance Category for the ETV CCEP

Projects conducted under the auspices of the ETV CCEP will meet or exceed the requirements of the American National Standards Institute/American Society for Quality Control (ANSI/ASQC), Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs, ANSI/ASQC E-4 (1994) standard. This protocol will ensure that the project results are compatible with and complementary to similar projects. ETV CCEP HVLP Coating Equipment TQAPPs will be adapted from this standard and the ETV Program Quality Management Plan (QMP). These TQAPPs will contain sufficient detail to ensure that measurements are appropriate for achieving project objectives, that data quality is known, and that the data are legally defensible and reproducible.

1.3 Logic and Organization of the Protocol Document

This HVLP coating equipment protocol document contains the sections outlined in the ANSI/ASQC E-4 standard. As such, this protocol identifies processes to be used, test and quality objectives, measurements to be made, data quality requirements and indicators, and procedures for recording, reviewing, and reporting data. Section No. 1 Revision No. 1 11/17/99 Page 2 of 47

The major technical sections to be discussed in this protocol are as follows:

- Project Description
- Project Organization and Responsibilities
- Quality Assurance (QA) Objectives
- Site Selection and Sampling Procedures
- Analytical Procedures and Calibration
- Data Reduction, Validation, and Reporting
- Internal Quality Control Checks
- Performance and System Audits
- Calculation of Data Quality Indicators
- Corrective Action
- Quality Control Reports to Management
- References
- Appendices.

1.4 Formatting

In addition to the technical content, this protocol also contains standard formatting elements required by the ANSI/ASQC E-4 standard and Concurrent Technologies Corporation (*CTC*) deliverables. Standard format elements include, at a minimum, the following:

- Title Page
- TQAPP Approval Form
- Distribution List
- Table of Contents (with an explanation of any deviations from Category II required elements)
- Document Control Identification (in the plan header):

Section No.	
Revision No.	
Date:	
Page:	of

1.5 Approval Form

Key ETV CCEP personnel will indicate their agreement and common understanding of the project objectives and requirements by signing the TQAPP Approval Form for each piece of equipment tested. Acknowledgment by each key person indicates commitment toward implementation of the plan. Figure 1 shows the Approval Form format to be used.

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Date Submitted:	QTRAK No.:	
Revision No.: Pro	oject Category:	
Title:		
Project/Tack Officer:		
EPA/Address/Phone No.:		
Interagency		
Agreement No.: Task	No.: Duration	on
APPROVALS		
APPROVALS CTC Project/Task Manager	Signature	Date
APPROVALS <i>CTC</i> Project/Task Manager <i>CTC</i> QA Officer	Signature	Date Date
APPROVALS <i>CTC</i> Project/Task Manager <i>CTC</i> QA Officer NRMRL/APPCD Project/Task Officer	Signature Signature Signature	Date Date Date

Figure 1. Testing and Quality Assurance Project Plan Approval Form

2.0 PROJECT DESCRIPTION

2.1 General Overview

Organic finishing processes are used by many industries for the protection and decoration of their products. Organic coatings contribute nearly 20 percent of total stationary area source volatile organic compound (VOC) emissions, as well as a significant percentage of air toxic emissions. Coating application equipment is continually being developed or redesigned to reduce any detrimental effects to the environment. This is primarily accomplished by increasing the transfer efficiency (TE) of the coating operation and, therefore, reducing the amount of coating used, (i.e., less overspray) and VOCs released into the environment. Often these coating equipment technologies are slow to penetrate the market because potential users, especially an ever-growing number of small companies, do not have the resources to test the new equipment in their particular application and may be constructively skeptical of the equipment provider's claims. If an unbiased, third-party facility could provide pertinent test data, environmentally friendly coating equipment technologies would penetrate the industry faster and accelerate environmental improvements.

The ETV CCEP, a joint venture of the U.S. Environmental Protection Agency (EPA) and *CTC* of Johnstown, Pennsylvania, in conjunction with the National Defense Center for Environmental Excellence (NDCEE) Program, has been established to provide unbiased, third-party data. The ETV CCEP has been tasked to develop, and subsequently utilize, a series of standardized protocols to verify the performance characteristics of coatings and coating equipment. This protocol will verify the performance of HVLP coating equipment that applies coatings to metal substrates.

To maximize the ETV CCEP's exposure to the coatings industry, the data from the verification testing will be made available on the Internet at the EPA's ETV Program website (*http://www.epa.gov/etv/*) under the P2 Innovative Coatings and Coating Equipment Pilot, as well as through other sources (e.g., publications, seminars). This will help establish the ETV CCEP's reputation in the private sector. A long-range goal of this initiative is to become a vital resource to the industry and, thus, self-sustaining through private support. This is in addition to its primary objective of improving the environment by rapidly introducing more environmentally friendly coating technologies into the industry.

2.1.1 Demonstration Factory Testing Site

CTC has been tasked under the NDCEE Program to establish a demonstration factory capable of prototyping processes that will reduce or eliminate environmentally harmful materials used or produced in manufacturing. To accelerate the transition of environmentally friendly processes to the manufacturing base, *CTC* offers the ability to test processes and products on full-scale, commercial equipment. This demonstration factory is a major national asset. It includes a combination of coating, cleaning, stripping, inorganic finishing, and recycle/recovery equipment. The coating equipment in the demonstration factory will be available for the pilot-scale testing performed in this project, (e.g., surface pretreatment, powder coating, electrocoating, wet spray, and conventional and infrared cure ovens). Ancillary equipment from plating, nonhalogenated cleaning, and nonchromate conversion coating are also available. Layouts of the *CTC* Demonstration Factory and the organic finishing line are shown in Figures 2 and 3, respectively.



Figure 2. CTC Demonstration Factory Layout



Figure 3. Demonstration Factory Organic Finishing Line

In the event that a particular technology demonstration or laboratory analysis cannot be performed at *CTC*, arrangements will be made to ensure the requirements of the TQAPP and all associated QA procedures are completed.

2.1.2 Laboratory Facilities

In support of the demonstration factory coating processes, *CTC* maintains extensive, state-of-the-art laboratory testing facilities. These laboratory facilities are used for the measurement and characterization of processes and specimens, as well as for bench-scale coating technology evaluations. Table 1 lists the various testing and evaluation laboratories and the representative equipment holdings that are relevant to ETV CCEP equipment projects.

Laboratory	Focus	Laboratory Equipment
Environmental Testing	 Identification and quantification of biological, organic, and inorganic chemicals and pollutants to all media. Industrial process control chemical analysis. 	Hewlett Packard 5972A GC/MS Varian Liberty 110 Sequential ICP P-E 4100ZL Graphite Furnace Mitsubishi GT06 Autotitrator P-E Headspace GC/ECD/FID TOC/Flashpoint/pH/Conductivity Graseby 2010 Isokinetic Stack Analyzer Graseby 2800 VOST Stack Sampler Questron Q-Wave 1000 Microwave Leeman PS200/AP200 Mercury Stations Millipore TCLP/ZHE Extraction Station Lachat Quickchem Flow Injection Analyzer
Destructive and Nondestructive Evaluation	Evaluation of product and process performance, and surface cleanliness.	Optically Stimulated Electron Emission X-ray/Magnetic/Eddy Current Thickness Salt Spray Corrosion Chamber Microhardness/Tensile/Fatigue/Wear
Materials and Mechanical Testing	Measurement of service and processing material and mechanical properties.	Noran and CAMScan Electron Microscopes Leco 2001 Image Analysis System Nikon and Polaroid Light Optical Microscopes EDAX Energy Dispersive Spectrometer Single Crystal Imaging Metallography Polishing/Grinding/Etching MTS Machines Tinius Olsen Testers Impact Testers
Powder Metallurgy	Investigation of powder properties.	Horiba LA900 Laser Particle Size Analyzer Autopore II 9020 Mercury Porosimeter Accupyc 1330 Pycnometer Gemini II 2370 Surface Area Analyzer
Intelligent Processing of Materials	Development and evaluation of embedded process sensors.	TEC Model 1600 Stress Analyzer Spectraphysics Argon & ND:YAg Lasers Resonance Frequency System
Risk & Environment Analysis	Management, monitoring, and evaluation of material and process alternatives from health and safety perspective.	Biosym: molecular modeling software MOPAC, Extend, HSC Chemistry, Riskpro, Sessoil, GIS
Calibration Laboratory	Calibration of equipment, sensors, and components to nationally traceable standards.	Transmation Signal Calibrator (milliamps, millivolts) Thermacal Dry Block Calibrator (Temperature) Druck Pressure Calibrator (Pressure) Fluke Digital Multimeter (Voltage)

 Table 1. Testing Laboratories and Representative Laboratory Equipment Holdings

2.1.3 ETV CCEP Technology Selection and Test Plan Development

A schematic diagram of the verification process is shown in Figure 4, and the following tasks planned for this project are listed below:

- Stakeholders' meeting investigates/identifies/prioritizes focus areas
- Identify technology providers
- Develop technology solicitation Commerce Business Daily (CBD) Announcement/Request for Technologies (RFT)
- Issue technology solicitation
- Review responses to solicitation
- Review generic protocol by stakeholders and technology providers
- Develop and obtain EPA approval of generic protocol document
- Obtain technology providers' commitment to program participation
- Provide TQAPPs for each piece of coating equipment tested
- Obtain technology provider concurrence with the TQAPP
- Obtain CTC and EPA approval of TQAPPs
- Conduct verification test of each HVLP gun
- Conduct CAS baseline tests of each HVLP vendor-supplied coating
- Prepare and provide draft test reports to EPA
- Prepare and provide final test reports to EPA
- Verification Statements issued by CTC.

Table 2 describes the general guidelines and procedures that will be applied to each TQAPP.

Table 2. Overall Guidelines and Procedures to be Applied to the TQAPP

- A detailed description of each part of the test will be given. This will include a detailed Design of Experiments and a schematic diagram of testing to be performed.
- Critical and noncritical factors will be listed. Noncritical factors will be held constant throughout the testing. Critical factors will be listed as control (process) factors or response (coating product quality) factors (see Section 2.2.12).
- The TQAPP will identify the testing site.
- Regardless of where the testing is performed, the ETV CCEP will ensure that the integrity of third-party testing is maintained.
- Regardless of where the testing is performed, the QA portion of the Generic Protocol will be strictly adhered to.
- A statistically significant number of samples will be analyzed for each critical response factor (see Table 5). Variances (or standard deviations) of each critical response factor will be reported for all results.

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Figure 4. Schematic of the Verification Process

HVLP Coating Equipment – Generic Testing and Quality Assurance Protocol

2.2 Technical/Experimental Approach and Guidelines

2.2.1 Description of HVLP Technology

The overall objective of the ETV CCEP is to verify pollution prevention characteristics and/or performance of coatings and coating equipment technologies, and to make the results of the verification tests available to prospective technology users. The ETV CCEP hopes to increase the use of more environmentally friendly technologies in product finishing, thereby reducing emissions. The objective of this particular protocol is to verify the performance of HVLP coating application equipment, and during the initial phase of this project, determine the relative transfer efficiency (TE) improvement of HVLP guns over conventional air spray (CAS) guns. Analysis methods used for these tests will follow those developed by the American Society for Testing and Materials (ASTM) or similarly accepted methods.

HVLP spray equipment is divided into two main categories—turbine and conversion. The turbine HVLP spray guns use a turbine compressor to generate large volumes of low-pressure air that is fed to the spray gun. The turbines are designed so that the input air pressure is consistently below 10 psig. The HVLP turbine compressor intrinsically transfers heat to the atomizing air that is supplied to the spray gun, which helps atomize paints that have a high viscosity. Turbine guns primarily use pressure-, or force-feed, systems to deliver the paint to the gun. Force-feed systems consist of a pressure pot that contains a drawtube that travels from the bottom of the pressure pot to the connection that leads to the spray gun. Air pressure above the coating forces the paint up through the drawtube, through the supply lines, and to the spray gun. A constant paint flow rate is achieved by maintaining constant air pressure to the delivery system. A variation of the pressure-feed spray gun uses a pressurized cup that is attached to the gun similar to the siphon-feed system described below.

Conversion HVLP spray guns use the existing high-pressure air supply that non-HVLP spray guns use. Conversion guns convert the low volume of air supplied at high pressure to a larger volume of air at lower pressure. Conversion HVLP spray guns use three types of paint delivery systems. First, force-feed systems, which are described above. Second, gravityfeed systems consist of a cup mounted on top of the spray gun. Hydrostatic pressure, as a result of gravitational forces, is the driving force behind the paint flow to the spray gun. As the volume of paint in the gravity cup decreases, the paint flow rate decreases. And third, siphon-, or suction-feed systems consist of a cup attached to the bottom of the spray gun, located near the air cap. The siphon cup contains a drawtube that leads from the bottom of the cup to the connection with the spray gun. The air pressure passing through the spray gun creates a negative pressure in the drawtube, drawing the paint up toward the spray gun. Higher viscosity paints require higher pressures through the spray gun to induce paint flow.

Compared to CAS guns, both HVLP gun types increase the average paint droplet size and reduce the velocity of the paint particles discharged from the gun. The increased droplet size, combined with the reduced velocity of the droplets, reduces the amount of overspray. Overspray is the small paint droplets that are carried around the product or the large paint droplet that bounce off the product because of the high velocity of the air currents or paint droplets. If more of the paint sprayed is actually applied to the product, less paint will be required to finish the work. The paint transfer characteristics of HVLP guns have been discussed in a number of published articles and reports (see References 1 through 5).

The amount of paint solids applied to the product divided by the amount of paint solids sprayed is known as transfer efficiency (TE). TE will be the primary criteria for verifying the performance of HVLP coating equipment in terms of pollution prevention. As the TE increases, less coating material is needed, reducing solvent emissions and the amount of paint solids that are released into the environment; therefore, coating equipment that is capable of achieving a higher TE provides a pollution prevention benefit to the end users.

The potential pollution prevention benefits of HVLP have encouraged regulators to require that end users only use equipment that is capable of meeting or exceeding the transfer efficiency of HVLP spray guns. HVLP was defined by the California South Coast Air Quality Management District Rule 1151 on June 13, 1997 as:

Equipment used to apply coatings by means of a spray gun, which is designed to be operated and which is operated between 0.1 and 10 pounds per square inch gauge (psig) air pressure measured dynamically at the center of the air cap and at the air horns.

This definition does not take into account input pressure or finish quality. The ETV CCEP was informed by several HVLP manufacturers that there are spray guns currently being marketed as HVLP which are slightly modified CAS guns operated at very low input air pressures. It is unlikely that these spray guns will provide a good quality finish when operated at output pressures less than 10 psig. To obtain the desired finish, the end user may increase the air pressure supplied to the gun, increasing the output pressure beyond 10 psig, and negating the environmental benefits

of the technology. If HVLP spray guns are to be used as they were intended, it is advantageous to verify that the equipment can provide the end user with an acceptable finish while operating the equipment as designed; therefore, this program will verify that equipment being marketed as HVLP will provide the user with an acceptable quality finish, while operating under the current definition of HVLP. The primary criteria for verifying the performance of specific types of HVLP coating equipment will be:

- Does the equipment meet the regulatory definition of HVLP, thereby providing a potential environmental benefit?
- What finish quality does the equipment provide while operating under the definition of HVLP?
- Does the equipment provide better transfer efficiency than the CAS baseline, thereby providing an actual environmental benefit?

From information gained during the testing of HVLP spray guns, the end users may better determine whether a particular HVLP spray gun would provide them with a pollution prevention benefit while meeting the finish quality requirements of their application. The end users must make an informed decision based on the best available data. This project intends to supply the end users with the unbiased technical data to assist them in that decision-making process.

The quantitative pollution prevention benefit in terms of improved TE depends on any of the innumerable factors that are unique to each coating production line. The task of verifying every possible combination of these factors is nearly impossible, and a test plan designed from a selection of these factors will provide data that is only representative of the exact conditions tested; however, in an effort to qualify the existence of an environmental benefit, this project will conduct a test to determine a qualitative transfer efficiency comparison of HVLP guns with respect to a CAS baseline.

2.2.2 Test Approach

The following approach will be used in the test protocol:

- Performance parameters to be verified are determined
- A standard test panel has been chosen that will enable thorough testing of coating equipment performance
- Required specifications for test coatings will be given to the equipment providers

- Equipment providers will supply all necessary equipment, the optimum HVLP equipment settings, the test coating, and a test coating reference panel (see Section 2.2.6)
- The ETV CCEP will complete a CAS baseline for TE and finish quality comparisons, as needed (see Section 2.2.7)
- A statistically valid test program that efficiently accomplishes the required objectives will then be used to analyze the results of these tests.

2.2.3 Standard Test Panel

The default standard test panel to be used for this project is a flat, coldrolled steel panel from ACT Laboratories, Inc., as shown in Appendix C, *Standard Test Panel*. The cold-rolled steel meets Society for Automotive Engineers (SAE) 1008 specifications. The test panel is 12 inches long, 4 inches wide, and made of 22-gauge steel. The panel is received unpolished and untreated. It has a ¼-inch hole punched in one end to suspend it from a hook.

The test panels will first undergo a pretreatment in preparation for the coating. The pretreatment portion of the Organic Finishing Line in the *CTC* Demonstration Factory is a seven-stage operation. The standard test panels will receive an alkaline cleaning followed by a deionized (DI) water rinse. Zinc phosphate is then applied, followed by another DI water rinse. A nonchromate sealer is then applied, followed by another DI water rinse. The pretreatment concludes with a dry-off stage. Prior to being coated, one random test panel per run will be removed for pretreatment analysis. During the pretreated with each run of 24 panels. These additional panels will be used as setup panels for the test and to fill in the blank space left on the racks due to the pretreatment analysis; therefore, a run will coat 23 standard test panels used to determine TE and finish quality, and one pretreated panel will be used only to calculate TE.

The Organic Finishing Line in the Demonstration Factory at *CTC* can handle parts up to 1.2 m x 1.2 m x 0.9 m (4 ft x 4 ft x 3 ft), weighing up to 113 Kg (250 lbs). Although designed to pretreat metal, the Organic Finishing Line is capable of applying special pretreatments needed for the particular substrates tested. Tests using other methods of pretreatment or other parts can be performed at the request of the equipment provider. For example, HVLP equipment providers may choose to target the wood finishing or non-metal coating industries, in which case, the test panels may be made of a substrate other than cold-rolled steel. The equipment providers will be responsible for funding additional tests. The remainder of this Generic HVLP Equipment Protocol will discuss the handling and

processing procedures to be used with the default standard test panels. The procedures used for alternate substrates may vary from this protocol. In these instances, the test panels to be used will be detailed in the associated TQAPP.

2.2.4 Coating Specification

Each equipment provider shall supply the test coating of its choice. The chosen coating shall meet the requirements specified in Appendix D, *Coating Specifications*. Each equipment provider will supply the ETV CCEP with a sufficient amount of coating to complete all tests associated with the HVLP gun being tested, the exact coating preparation instructions, and the test coating supplier's reference panel prepared using a CAS gun. At an additional charge, the equipment providers may request that further coating quality tests be performed using other coatings.

2.2.5 Standard Apparatus

This protocol includes a standard apparatus setup for all tests associated with the verification of HVLP spray guns. Appendix A, *Apparatus Setup*, shows the selected apparatus setup, and Appendix B, *Equipment Testing Location*, shows the testing location relative to the Organic Finishing Line. The testing will be performed in one of the liquid spray booths in the *CTC* Demonstration Factory.

The standard test panels will be transported through the system on racks suspended from the overhead conveyor. A rack will hold up to eight test panels in a single row, as shown in Appendix A. The test panels will be fixtured on the rack to minimize movement during spraying. The rack consists of a flat bar that connects the hooks to minimize side-to-side rotation of the panels and a second bar that prevents the bottom of the panels from moving away from the gun. A mechanical stop mechanism will align the racks of test panels in the proper position relative to the spraying mechanism. Once the racks are in position, the spraying mechanism's programmable logic controller (PLC) will activate the motors that drive the linear motion translators. The translators will move both horizontally and vertically. This setup will be able to cover an area approximately 1.37 m x 1.37 m (4.5 ft x 4.5 ft). The panels will be automatically sprayed using vertical overlap of the spray pattern. The spraying mechanism's PLC will also control the triggering of the HVLP spray gun by way of a pneumatically actuated cylinder. During dwell time between passes, paint flow will be interrupted to minimize paint usage. Once the spray application is complete, the mechanism's PLC will release the mechanical stop holding the rack so that the overhead conveyor can move the next rack into position.

The HVLP gun will be mounted on the translator by clamping it to an arm that extends from the vertical translator's carrier plate. A pneumatic cylinder, which is controlled by the translator's PLC, will pull the trigger on command.

2.2.6 Test Coating Reference Panel

The HVLP equipment provider will supply the coating reference panel for the test coating of its choice, unless the selected coating has already been used for an HVLP equipment verification test. The coating reference panel must be prepared with CAS equipment by representatives of the test coating manufacturer. The coating reference panel must meet the target ranges established in the associated TQAPP and represent the ideal coating finish that was intended by the test coating manufacturer. The coating reference panel will be analyzed for dry film thickness (DFT), distinctness-of-image (DOI), gloss, and visual appearance. The panels coated by the HVLP spray guns during the verification test will be compared to the coating reference panel on the basis of these four finish quality analyses. These comparisons will indicate whether the HVLP spray gun is able to apply an acceptable quality finish. If the coating reference panel does not meet the finish quality target ranges established in the associated TQAPP, the CAS baseline test will be used for both the TE and finish quality comparisons.

2.2.7 CAS Baseline

A CAS baseline test will be performed for each new coating that is used during the verification of HVLP spray guns. The CAS baseline will be used to determine the relative improvement in TE that the HVLP can provide. The CAS baseline panels will also be evaluated for DFT, gloss, and visual appearance. The DOI analyses will not be performed on the CAS baseline panels as part of their finish quality evaluation unless the test coating reference panel fails to meet the four finish quality target ranges. Although the CAS baseline panels are not the finish quality reference, the finish quality evaluation of the CAS baseline panels is intended to show that the finish quality of the coated test panels was not sacrificed to obtain a higher TE.

The CAS baseline will consist of three CAS guns having the same fluid delivery system as the HVLP spray gun being verified. CAS baseline testing will be designed and performed by the ETV CCEP personnel. Certain operating parameters used for the CAS baseline will be identical to the parameters used for the HVLP verification test. For example, the CAS guns will be operated in the same apparatus setup as the HVLP spray gun, the CAS gun fan pattern at the target will be the same size as the fan Section No. 2 Revision No. 1 11/17/99 Page 16 of 47

> pattern used during the HVLP test, the CAS gun atomization characteristics will be similar to the HVLP test, and the same test coating will be used and mixed according to the same proportions. Other parameters will be developed from the CAS gun or coating manufacturer's recommendations and/or experimental trials performed by the ETV CCEP. The distance to target may vary between the CAS baseline and the HVLP test, the fan and atomization adjustments will be specific to each gun, the atomizing air pressure will be determined for each gun to meet the fan pattern, and the horizontal gun traverse speed will be determined for each gun to obtain the appropriate DFT.

> During the experimental trials performed by the ETV CCEP to obtain the CAS gun-specific parameters, several three-panel sets will be coated using the same application pattern, vertical drop, and flash time, as the HVLP test. Each three-panel set will be coated using different horizontal gun speeds. The trial-and-error method will be used to achieve a DFT comparable to the HVLP test. The panel sets will be cured using the same procedure as the HVLP test. After they are cured, the average DFT of each set of panels will be determined. If none of the average DFTs for the panel sets are within the target range, the range of application speeds will be adjusted, and additional sets of panels will be coated. This process will be repeated until a speed is identified that provides a DFT similar to that obtained for the HVLP test. Once the appropriate horizontal gun traverse speed is identified, that speed will be entered into the CAS TQAPP and used for the baseline test. The operating parameters for each of the three CAS guns will be determined in the same manner.

2.2.8 Design of Experiment

A mean value and variance (or standard deviation) will be reported for each critical response factor. If a technology provider wants to verify a particular performance characteristic, he or she will be asked to submit a confidence limit and specification limit (acceptable quality limit) for that characteristic. If the technology provider does not submit a confidence and specification limit, a default 95% confidence limit will be applied.

Any characteristic the equipment provider wants to have verified will be included in the experimental design. The appropriate number of test panels to be coated and analyzed will be based on the confidence limit, specification limit, and the appropriate statistical test to be applied to the results (e.g., Student's T-Test, Chi Square Test, or F-Test).

2.2.9 Performance Testing

The ETV CCEP will provide the equipment providers with key noncritical factors to be used for testing, including the requirements that must be met by the test coating that the equipment provider chooses (see requirements in Appendix D). The equipment providers will then supply the ETV CCEP with the appropriate equipment settings for their coating, such as cap pressure, input air pressure, gun traverse speed, and paint flash/dwell time. No effort will be made by the ETV CCEP during the actual test runs to optimize the equipment, although the equipment providers will be afforded the opportunity to do so during the startup phase of the testing.

The HVLP verification and CAS baseline tests will consist of five (5) runs with three (3) racks of eight (8) panels in a single row. This will enable both total and lot-to-lot variation to be determined for each response factor. The statistical analyses for all response factors will be carried out using Minitab statistical software.

Before each test, a set of dummy panels will be coated to ensure that the equipment parameters are set correctly (the equipment provider may wish to assist in this step). For the TE portion of the tests, the weight of each panel used to determine TE will be measured between the pretreatment and coating operations and again after the coated panels are cured. The input air pressure will be monitored throughout the test, and the air pressure at the cap and air horns will be measured using a verified test cap before each HVLP test. The paint flow, from the pressure-feed fluid delivery systems only, will be measured by an in-line flow meter.

The spray booth air filters will be changed prior to setting up the standard apparatus for each gun. The pressure drop across the filters will be checked prior to each run and at the end of the test. The pressure drop is monitored in the event that the filter bank system malfunctions. A pressure drop across the filter bank greater than 1 cm (0.4 in.) of water shall indicate that the system requires service. As a comparison, the spray booth air filter will also be changed before each CAS gun is set up and tested as part of the TE baseline. This will ensure that the difference in the initial booth air velocity between the guns is minimized. The booth air velocity will be measured in close proximity to the panels. Although the air velocity through the booth exceeds 0.5 m/s (100 ft/min), the velocity measured near the panels may be lower due to the disruption of the air currents by the rack of panels.

Standard test panels will be used to measure equipment performance. The test panels will be used for DFT, gloss, DOI, TE, and visual appearance analyses. The coating characteristics may be affected by other parameters of the testing process, such as pretreatment, apparatus setup, and cleanup methods. Noncritical control factors will be monitored or held relatively

constant for the verification and CAS baseline tests. DFT measurements will be used to determine the variations in film thickness; gloss and DOI will be used to analyze the quality of the coating finish; and TE measurements will be used to determine the qualitative comparison between CAS guns and HVLP spray guns. Finally, the visual appearance analysis will identify any abnormalities in the applied coating.

2.2.10 Quantitative Measurements

To verify that the equipment meets the definition of HVLP, a certified test cap will be used to determine the output pressure of each HVLP spray gun tested. The HVLP equipment providers will supply the certified test caps along with the other necessary spray equipment. The ETV CCEP will verify the accuracy of the test caps and/or obtain documentation verifying their accuracy.

The measurements for coating temperature and viscosity, and the samples used for coating density, VOC content, and percent solids, will be taken immediately before the coating is transferred to the fluid-feed system. The VOC content will be determined using ASTM D 3960, unless it can be documented that no exempt solvents or water are contained in the test coating. If this is the case, ASTM D 2369 may be used where all volatiles are assumed to be VOCs. The *CTC* work instructions for these measurements are found in Appendix F.

The DFT measurements will follow ASTM B 499 (Magnetic). Thickness measurements will be taken on the standard test panel as shown in Appendix C. Measurements using the magnetic method will be taken on each standard test panel coated. From this data, an overall DFT and a DFT variation across the standard test panel will be reported. The purpose of this comparison is to verify that a uniform DFT has been applied to the standard test panels.

The DOI analysis will approximately follow Method B of ASTM D 5767, except that the sliding combed shutter is replaced by a rotating, eightbladed disc.

The TE tests will follow Procedure A of ASTM D 5286, with the following exception—the weight measurements will not be performed at the paint booth. Instead, the minimum necessary equipment will be disconnected from the setup and transported to a calibrated laboratory balance. Every effort will be made to ensure that no coating is lost during this process. A TE value will be determined for each run and on a run-to-run basis.

The visual appearance analysis will use normal lighting to examine the surface of the painted panel. The panels will be examined for fish-eyes in the finish, the presence of orange peel, the evenness of the coating, and the difference in the visual gloss caused by sandpaper finish, drips, runs, and inclusions (such as dirt, fuzz, fibers, etc.). A comparison will be made from panel to panel, rack to rack, and run to run.

2.2.11 Participation

The Demonstration Factory at *CTC* provides a unique capability for demonstrating and evaluating full-scale manufacturing process applications. Full-scale processing and testing can be carried out on any of the process technologies within the Demonstration Factory without concern for the many problems associated with trying to do these same tests on manufacturing lines. Because of this existing capability, most of the tests and demonstrations will be performed at the Demonstration Factory. ETV CCEP technical staff will be responsible for performing all necessary tests and demonstrations required for performance evaluation and full-scale validation. Where specific equipment required for testing is not available, ETV CCEP will work with other facilities to perform the required work. For verification testing of HVLP equipment, it is anticipated that all tests will be completed at the *CTC* demonstration factory.

To help ensure proper equipment setup and operation, the equipment providers will be invited to participate in the startup phase of the testing and to observe the testing of their equipment.

2.2.12 Critical and Noncritical Factors

For the purposes of this protocol, the following definitions will be used for critical control factors, noncritical control factors, and critical response factors. A critical control factor is a factor that is varied in a controlled manner within the design of experiments matrix to determine its effect on a particular outcome of a system. Noncritical control factors are all the factors that are to be held relatively constant or randomized throughout the testing for each specific piece of equipment (some noncritical factors may vary from equipment to equipment). Critical response factors are the measured outcomes of each combination of critical and noncritical control factors given in the design of experiments.

In this context, the term "critical" does not convey the importance of a particular factor (that can only be determined through experimentation and characterization of the total process), but rather, its relationship within the design of experiments. In the case of the verification testing of a particular piece of coating equipment, there is only one critical control

factor, and that is the piece of coating equipment itself. All other processing factors will be held relatively constant (or randomized) and are noncritical control factors; therefore, the multiple runs and sample measurements within each run for each critical response factor will be used to determine the amount of variation expected for each critical response factor.

For all projects, the critical control factors, noncritical control factors, and critical response factors will be identified in a table format along with acceptance criteria (where appropriate), data quality indicators, measurement locations, and measurement frequencies, broken down by each trial or experiment. For example, for each HVLP spray gun design, parameters associated with metal surface pretreatment would remain constant and, thus, be noncritical control factors, and a parameter such as gloss would be identified as a critical response factor.

The only critical control factor is the coating equipment itself (see Table 3). The equipment provider's recommendations for the coating to be used, optimum input pressures, gun traverse speed, and other operating parameters will be followed. As a result, some noncritical control factors (see Table 4) will likely vary from one piece of coating equipment to another. The critical response factors that will be measured in these tests are given in Table 5.

Tables 3 through 5 below summarize the critical and noncritical factors that will be monitored throughout the testing. The values in the Total Numbers column are based on the default test scenarios.

Critical Control	Fluid	Fluid	Air	Fan
Factor	Tip	Needle	Cap	Pattern
Equipment Type and Model	from equipment provider	from equipment provider	from equipment provider	from equipment provider

Table 3. Critical Control Factors

			<u> </u>	1
Noncritical	Set Points/	Measurement		Total Number
Factor	Acceptance	Location	Frequency	for Each Test*
	Criteria			
Input Air Pressure	from equipment	Factory floor	Once per run	5
Gun/Pot	provider		-	
Products involved in	Standard Test	N/A	24 Standard	120 Standard
Testing	Panels		Test Panels	Test Panels
		ļ	per run	
Zinc Phosphate	2.1 - 2.7	Random panel	1 Standard	5
Pretreatment Weight	g/m²	removed prior to	Test Panel per	
	202.216^{-2}	the spray booth	run	1
Surface Area of Each	$303-316 \text{ cm}^2$	Top and right	I Standard	1
Panel Coated	(4/-49 m.)	edge of panel	Test Panel per	
Ambient Factory	< 60% RH	Retween booth	Once each run	5
Relative Humidity	< 0070 Km	and oven	Once caen run	5
Ambient Eastony	21.1.26.790	Detween booth	Once each min	5
Amplent Factory	21.1 – 26.7°C	and oven	Once each run	5
	500/ DH			
Spray Booth Relative	< 60% KH	Inside the wet	Once each run	5
Humidity		spray bootn		
Spray Booth	21.1 – 26.7°C	Inside the wet	Once each run	5
Temperature		spray booth		
Spray Booth Air	0.2-0.5 m/s	Inside the wet	Once per test	1
Velocity	(40-100 ft/min)	spray booth		
Distance to Panels	15.2-45.7 cm	Factory floor	Once per test	1
	(6-18 in.)			
Temperature of Panels,	21.1 – 26.7°C	Factory floor	Once per run	5
as Coated		-	-	
Horizontal Gun	from equipment	Factory floor	Once per test	1
Traverse Speed	provider			
Vertical Drop Between	from equipment	Factory floor	Once per test	1
Passes	provider		oner r	-
Dwell Time Between	from equipment	Factory floor	Once per test	1
Passes	provider	1 actory 11001	Once per test	1
VOC Content of	from coating	Sample from	1 sample each	5
Applied Coating	vendor	coating not	1 sample each	5
	Vendor		1,000	
Density of Applied	from coating	Sample from	1 sample each	5
Coating	vendor	coating pot	run	
% Solids of Applied	from coating	Sample from	1 sample each	5
Coating	vendor	coating pot	run	
Coating Temperature,	from coating	Sample from	1 sample each	5
as Applied	vendor	coating pot	run	
Coating Viscosity, as	from coating	Sample from	Before and	10
Applied	vendor	coating pot	after run	
Oven Temperature	from coating	Factory floor	Once each run	5
	vendor	-		
Oven Cure Time	from coating	Factory floor	Once each run	5
	vendor	-		

 Table 4. Noncritical Control Factors

* Based on the default test design.

Critical Response Factor	Measurement Location	Frequency	Total Number for Each Test ^b
Cap Air Pressure ^c	Cap and Air Horns	Once per test	1
Paint Flow Rate ^d	In-line after the fluid valve	Once per run	5
Total Paint Flow ^d	In-line after the fluid valve	Once per run	5
Overall Dry Film Thickness (Magnetic methods)	9 points in a lattice pattern on each coated face of the Standard Test panel	9 points on each of 5 Standard Test Panels per run, 5 runs	225
Dry Film Thickness Variation	Calculated from magnetic dry film thickness data	Variation on individual panels and variation from run to run	N/A
Gloss	from ASTM D 523	3 points on each of 5 Standard Test Panels per run, 5 runs	75
Distinctness of Image (DOI)	from ASTM D 5767 Test Method B ^e	3 points on each of 5 Standard Test Panels per run, 5 runs	75
Visual Appearance	Entire test panel and entire rack	1 per Panel, 1 per rack, and 1 per run	135
Transfer Efficiency (TE)	from ASTM D 5286	Once per run	5

Table 5. Critical Response Factors^a

^a See Sections 2.1.3 and 2.2.1 for the environmental basis on which these factors relate.

^b Based on the default test design.

•

^c HVLP spray guns only.

^d Pressure feed fluid delivery systems only.

^e Except that the sliding combed shutter is replaced by a rotating eight-bladed disc.

Qualitative noncritical control factors used in the verification tests include:

- Equipment Preparation
- Flash Time Between Coats
- from equipment provider from equipment provider
- Number of Passes fro
- Spray Pattern

- from equipment provider from equipment provider
- Target Dry Film Thickness
- from custom coating vendor.

2.3 Schedule

CTC uses standard tools for project scheduling. Project schedules are prepared in Microsoft Project or Primavera, which are accepted industry standards for scheduling. Project schedules show the complete work breakdown structure (WBS) of the project, including technical work, meetings, and deliverables. Each TQAPP will contain an estimated schedule for the verification testing activities.

3.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

CTC employs a matrix organization, with program and line management, to perform projects. The laboratory supports Project Managers and Technical Project Leaders by providing test data. Laboratory Analysts report to the Laboratory Manager. The Laboratory Manager coordinates with the Technical Project Leader on testing schedules. The Technical Project Leader is the conduit between the laboratory and the Project Manager. The Technical Project Leader answers directly to the Project Manager of a task. For the ETV CCEP, the Technical Project Leaders will be responsible for preparing the TQAPPs and the internal demonstration plans for each test.

Additionally, a QA Officer, who is independent of both the laboratory and the program, is responsible for administering *CTC* policies developed by the Quality Committee. These policies provide for and ensure that quality objectives are met for each project, and cover laboratory testing, factory demonstration processing, engineering decisions, and deliverables. The QA Officer reports directly to *CTC* senior management and is organizationally independent of project or program management.

The project organization chart, showing lines of responsibility and the specific *CTC* personnel assigned to this project, is presented in Figure 5. A summary of the responsibilities of each *CTC* participant, their applicable experience, and their anticipated time dedication to the project during testing and reporting is given in Table 6.



Figure 5. Project Organization Chart

Key CTC Personnel and Roles	Responsibilities	Applicable Experience	Education	Time Dedication for Phase
Dave Roberts NDCEE Program Director	Directs NDCEE Program. Accountable to <i>CTC</i> Technical Services Director and <i>CTC</i> Corporate Management.		BS Mechanical Engineering	5%
Brian Schweitzer Manager, Process Engineering/ Technical Project Manager	Responsible for overall ETV CCEP technical aspects, budget, and schedule issues on daily basis. Accountable to NDCEE Program Director.	Process Engineer (11 years) Project Manager, Organic Finishing (6 years)	BS Mechanical Engineering	50%
Jack Molchany QA Officer	Responsible for overall project QA. Accountable to NDCEE Program Director	QA/QC and Industrial Operations (12 years) Quality Management and ISO 9000 (6 years) Environmental Compliance and ISO 14000 Management Systems (6 years)	BS Industrial Engineering	5%
Rob Fisher Staff Process Engineer/ Technical Project Leader	Technical project support. Process design & development. Accountable to Technical Project Manager.	Organic Finishing Regulations (7 years)	BS Chemical Engineering P.E.	50%
Melissa Klingenberg Staff Process Engineer/ Technical Project Leader	Technical project support. Process design & development. Accountable to Technical Project Manager.	Process Engineer, Inorganic Finishing (7 years) Organic Finishing (2 year)	BS Chemistry/ Biology MS MSEP	50%
Herb Ashley Finishing Engineer/ Factory Operations Lead	Oversees day-to-day operation of Organic Finishing Line. Provides technical project support. Accountable to Technical Project Manager.	Organic Finishing Experience (28 years)		10%
Stephen Kendera Sr. Organic Finishing Technician	Performs day-to-day operations of the Organic Finishing Line. Accountable to Finishing Engineer	Industrial Paint and Coatings Experience (26 years)		10%
Dave James Process & Materials Characterization Manager/Laboratory Manager	Coordinates testing lab and technical data review. Accountable to Technical Project Manager, NDCEE Program Director.	Environmental Engineering (17 years) Project/People Management (17 years) ISO 9000/14000 Management Systems (5 years)	MS Environmental Engineering BS Ecology	<5%
Lynn Summerson Laboratory Leader	Laboratory analysis Accountable to Laboratory Manager	Industrial and Environmental Laboratory Testing (18 years)	MS Chemistry BS Chemistry	20%
Tammy Powers Associate Laboratory Leader	Laboratory analysis Accountable to Laboratory Manager	Environmental and Municipal Laboratory Testing (8 years)	BS Biology	10%
Brian Albright Assistant Laboratory Analyst/ Pretreatment Operator	QC Analysis Accountable to Laboratory Manager	Environmental and QC Testing (5 years)	BS Chemistry	10%

Table 6. Summary of ETV CCEP Experience and Responsibilities

The *CTC* personnel specified in Figure 5 and Table 6 are responsible for maintaining communication with other responsible parties working on the project. The frequency and mechanisms for communication are shown in Table 7.

Initiator	Recipient	Mechanism	Frequency
Program Director or Technical Project Manager	EPA Project Manager	Written Report Verbal Status Report	Monthly Weekly
Technical Project Manager	Program Director	Written or Verbal Status Report	Weekly
Laboratory Manager	Technical Project Manager	Data Reports	As generated
QA Officer	Program Director	Quality Review Report	As required
EPA Project Manager	CTC	On-Site Visit	At least once per year

 Table 7. Frequency and Mechanisms of Communications

Special Occurrence	Initiator	Recipient	Mechanism/ Frequency
Schedule or Financial Variances	Program Director or Technical Project Manager	EPA Project Manager	Telephone call, written follow-up report as necessary
Major (will prevent accomplishment of verification cycle testing) Quality Objective Deviation	Program Director or Technical Project Manager	EPA Project Manager	Telephone call with written follow-up report
4.0 QA OBJECTIVES

4.1 General Objectives

The overall objectives of this ETV CCEP protocol are to verify the performance of HVLP coating application equipment in providing a quality finish while operating under the conditions defined by law, to confirm the relative transfer efficiency improvement of HVLP guns over CAS guns, and to make the results of the testing available to prospective end users. These objectives will be met by controlling and monitoring the critical and noncritical factors, which are the specific QA objectives for this protocol. Tables 3 and 4 list the critical and noncritical control factors, respectively.

The analytical methods that will be used for coating evaluations are adapted from ASTM Standards, or equivalent standards. The QA objectives of the program and the capabilities of these test methods for product and process inspection and evaluation are synonymous because the methods were specifically designed for evaluation of the coating properties under investigation. The methods will be used as published, or as supplied, without major deviations. Minor deviations are noted in this protocol and will be noted in the subsequent TQAPPs. The specific methods to be used for this project are attached to this document as Appendix E, *ASTM Methods*.

4.2 Quantitative QA Objectives

Quality assurance parameters, such as precision, accuracy, and completeness, are presented in Tables 8 and 9. Table 8 presents the manufacturers' stated capabilities of the equipment used to measure noncritical control factors. The precision and accuracy parameters listed are relative to the true value that the equipment measures. Table 9 presents the precision and accuracy parameters for the measurement equipment for the critical response factors. Precision and accuracy are determined using duplicate analysis and known standards and/or spikes and must fall within the values found in the specific methods expressed.

The statistical support engineer, QA Officer, and laboratory personnel will coordinate efforts to calculate and interpret the test results.

Measurement	Method	Units	Precision	Accuracy	Completeness
Input Air Pressure	Pressure gauge	psig	<u>≤</u> 0.4	±5%	90%
Products involved in Testing	Standard Test Panels	# of panels	0	±0%	100%
Zinc Phosphate Pretreatment Weight	ASTM B 767	g/m ²	<u>≤</u> 0.01	±0.01	90%
Surface Area of Each Panel Coated	Ruler	cm (in.)	<u>≤</u> 0.3 (≤0.13)	<u>+</u> 0.2 (±0.06)	90%
Ambient Factory Relative Humidity	Thermal Hygrometer	RH	$\leq 6\%$ of full scale	±3% of full scale	90%
Ambient Factory Temperature	Thermal Hygrometer	°C	≤6% of full scale	±3% of full scale	90%
Spray Booth Relative Humidity	Thermal Hygrometer	RH	≤6% of full scale	±3% of full scale	90%
Spray Booth Temperature	Thermal Hygrometer	°C	≤6% of full scale	±3% of full scale	90%
Spray Booth Air Velocity	per ACGIH	m/s (ft/min)	<u>≤</u> 0.06* <u>(≤</u> 10)	<u>+0.03*</u> (+5)	90%
Distance to Panels	Ruler	cm (in.)	<u>≤</u> 0.3 (≤0.12)	<u>+</u> 0.2 (±0.06)	90%
Temperature of Panels, as Coated	IR Thermometer	°C	≤1.0%	±1.0%	90%
Horizontal Gun Traverse Speed	Stopwatch	cm/s (in./sec)	<u>≤</u> 0.002%	±0.001%	90%
Vertical Drop Between Passes	Ruler	cm (in.)	<u>≤</u> 0.3 (≤0.12)	<u>+</u> 0.15 (±0.06)	90%
Dwell Time Between Passes	Stopwatch	seconds	<u>≤</u> 0.002%	±0.001%	90%
VOC Content of Applied Coating	ASTM D 3960	g/l (lb/gal)	<u>≤</u> 1.2%	±1.8%	90%
Density of Applied Coating	ASTM D 1475	g/l (lb/gal)	≤1.2%	±1.8%	90%
% Solids of Applied Coating	ASTM D 2369	%	<u><</u> 3.0%	±4.7%	90%
Coating Temperature, as Applied	Thermometer	°C	≤1.0 °C	±0.2 °C	90%
Coating Viscosity, as Applied	ASTM D 1200	seconds	<u>≤</u> 20%	±10%	90%
Oven Temperature	Thermocouple	°C	<u>≤</u> 4.4 °C	±2.2 °C	90%
Oven Cure Time	Stopwatch	minutes	<u>≤</u> 0.002%	±0.001%	90%

Table 8. QA Objectives for Precision, Accuracy, and Completeness for All
Noncritical Control Factor Performance Analyses

ACGIH - American Conference of Governmental Industrial Hygienists, Inc.

st Accuracy and Precision stated by the manufacturer for velocities ranging from 20-100 ft/min

Measurement	Method	Units	Precision	Accuracy	Completeness
Test Cap Air Pressure⁺	Equipment specifications	psig	(1)	(1)	90%
Paint Flow Rate ⁺⁺	Flow meter	cm ³ /min	<u>≤</u> 0.5%	±0.5%	90%
Total Paint Flow ⁺⁺	Flow meter	cm ³	≤0.5%	±0.5%	90%
Dry Film Thickness (DFT) Magnetic	ASTM B 499	mils ⁽²⁾	<u>≤</u> 20%	10% true thickness	90%
DFT Variation	N/A	N/A	N/A	N/A	N/A
Distinctness of Image (DOI) ⁽³⁾⁺	ASTM D 5767 Method B	DOI Units	<u>≤</u> 20%	±3 DOI units	90%
Gloss	ASTM D 523	gloss units	≤20%	±1 gloss unit	90%
Visual Appearance	N/A	N/A	N/A	N/A	N/A
Transfer Efficiency (TE)	ASTM D 5286 Test Method A	%	≤25% ⁽⁴⁾	$rsd \le 20\%^{(4,5)}$	90%

Table 9. QA Objectives for Precision, Accuracy, and Completeness for AllCritical Response Factor Performance Analyses

(1) To be provided by HVLP vendor

(2) 1 mil = 0.001 inch

(3) Performed by ACT Laboratories, Inc.

(4) Unknown according to ASTM D 5286

(5) rsd =relative standard deviation

+ HVLP spray guns only

++ Pressure-feed fluid delivery systems only

N/A = Not Applicable

4.2.1 Accuracy

Standard reference materials, traceable to national sources, such as the National Institute for Standards and Technology (NIST) for instrument calibration and periodic calibration verification, will be procured and used where such materials are available and applicable to this project. For reference calibration materials with certified values, acceptable accuracy for calibration verification will be within the specific guidelines provided in the method if verification limits are given; otherwise, 80%–120% of the true reference values will be used (see Tables 8 and 9). Reference materials will be evaluated using the same methods as for the actual test specimens.

4.2.2 Precision

The experimental approach of this protocol specifies the exact number of test panels to be coated. The analysis of replicate test panels for each coating property at each of the experimental conditions will occur by design. The degree of precision will be assessed based on the agreement of all replicates within a property test group.

4.2.3 Completeness

The laboratory strives for at least 90 percent completeness. Completeness is defined as the number of valid determinations expressed as a percentage of the total number tests conducted, by test type.

4.2.4 Impact and Statistical Significance Quality Objectives

All laboratory analyses will meet the accuracy and completeness requirements specified in Tables 8 and 9. The precision requirements also should be achieved; however, a nonconformance may result from the analysis of replicates due to limitations of the coating technology under evaluation, and *not* due to processing equipment or laboratory error. Regardless, if any nonconformance from TQAPP QA objectives occurs, the cause of the deviation will be determined by checking calculations, verifying the testing and measuring equipment, and performing a reanalysis. If an error in analysis is discovered, reanalysis of a new batch for a given trial will be considered, and the impact to overall project objectives will be determined. If the deviation persists despite all corrective action steps, the data will be flagged as not meeting the specific quality criteria, and a written discussion will be generated.

If all analytical conditions are within control limits and instrument and/or measurement system accuracy checks are valid, the nature of any nonconformance may be beyond the control of the laboratory. If, given that laboratory quality control data are within specification, any nonconforming results occur, the results will be interpreted as the inability of the coating equipment undergoing testing to produce parts meeting the performance criteria at the given set of experimental conditions.

4.3 Qualitative QA Objectives: Comparability and Representativeness

4.3.1 Comparability

Organic coating technologies will be utilized and/or operated at vendorrecommended conditions or conditions otherwise established in agreement with the project stakeholders for each protocol. The data will be Section No. 4 Revision No. 1 11/17/99 Page 32 of 47

> comparable from the standpoint that other testing programs could reproduce similar results using a specific TQAPP. Coating and environmental performance will be evaluated using EPA, ASTM, and other industry-wide or nationally accepted testing procedures as noted in previous sections of this protocol. Process performance factors will be generated and evaluated according to standard best engineering practices. In addition, suppliers will be asked to provide performance data for their product and the results of preliminary or prior testing relevant to this protocol, if available.

Test specimens generated at *CTC* will be compared to these performance data and to the other applicable end user and industry specifications. These performance standards will be used to verify the performance of the technology. Additional assurance of comparability will be derived from the routine use of precision and accuracy indicators as described above, the use of standardized and accepted methods, and the traceability of reference materials.

4.3.2 Representativeness

The limiting factor to representativeness is the availability of a large sample population. Experimental designs will be constructed such that projects will either have sufficiently large sample populations per trial or statistically significant fractional populations. The tests will be conducted at the paint and equipment supplier-recommended operating conditions. If the test data meet the quantitative QA criteria (precision, accuracy, and completeness), the samples will be considered representative of the coating technologies under evaluation and used to interpret the outcomes relative to the specific project objectives.

4.4 Other QA Objectives

There are no other QA objectives as part of this evaluation.

4.5 Impact of Quality

Due to the highly controllable nature of the test panel evaluation methods and predictability of factors affecting the quality of the laboratory testing of panels, the quality control of test panel qualifications is expected to fall within acceptable levels. Comparison of response factors will be checked for run-to-run process variations.

5.0 SITE SELECTION AND SAMPLING PROCEDURES

5.1 Site Selection

This project will be executed at *CTC*'s facilities in Johnstown, Pennsylvania, and *CTC* personnel will perform all processing and testing. The site for application and evaluation will be at *CTC* in the Demonstration Factory at the Environmental Technology Facility (ETF) under the direct control of the Engineering and Statistical Support and Organic Finishing Line Groups. Analysis will be performed in the *CTC* Testing Laboratory at the ETF by the Environmental Laboratory.

5.2 Site Description

Figure 2, in Section 2.1.1, illustrates the overall layout of the Demonstration Factory and the location of the process equipment that may be used for this protocol. The testing in this project involves the use of the pretreatment process with an associated dry-off oven, the liquid spray booths, and the wet cure oven. Other equipment or testing sites may be used as necessary.

5.3 Sampling Procedures and Handling

Standard test panels will be used in this project. These will be pre-labeled by stamping them with a unique alphanumeric identifier. The number of test panels processed during the testing depends on the experimental design, which in turn, depends on any equipment provider's claim(s) about performance characteristics and the respective confidence levels given in the responses to the RFT. If no specific performance characteristics are requested for verification by the HVLP equipment providers, the default experimental design will then be used. The experimental design uses 120 samples for the TE test (5 runs with 3 racks per run and 8 panels per rack).

A factory operations technician and laboratory analysts will process the test panels according to a preplanned sequence of stages, which includes those identified in Table 10.

Procedure	Operation s Technician	Laboratory Analyst
1. Numbering of the panels		Х
2. Shot-blast the panels	Х	
3. Pretreatment panels with zinc phosphate		X
4. Initial weight of panels		X

Table 10. Process Responsibilities

5. Remove 1 panel/run for pretreatment		X
analysis		
6. Arrange panels on the racks		Х
7. Prepare the coating	Х	X
8. Set up the HVLP gun	Х	
9. Take coating samples and measurements		Х
10. Load coating in the fluid system and prime	Х	
gun		
11. Perform set up trials (before first run only)	Х	
12. Initial weight of fluid system and/or gun	Х	Х
13. Apply coating to the panels	Х	
14. Take process measurements		Х
15. Cure the panels	Х	
16. Wrap and stack panels for transfer to the lab	X	

A laboratory analyst will record the date and time of each run and the time each measurement was taken. When the panels are removed from the racks, they will be separated by a layer of packing material and stacked for transport to the laboratory. The laboratory analyst will process the test panels through the laboratory login prior to performing the required analyses.

5.4 Sample Custody, Storage, and Identification

The test panels will be delivered to the laboratory for login and given a unique laboratory ID number. The analyst delivering the test panels will complete a custody log indicating the sampling point IDs, sample material IDs, quantity of samples, time, date, and analyst's initials. The product evaluation tests will also be noted on the custody log. The laboratory's sample custodian will verify this information. Both the laboratory analyst and sample custodian will sign the custody log to indicate transfer of the samples from the coating processing area to the laboratory analysis area. The laboratory sample custodian will log the test panels into a bound record book, store the test panels under appropriate conditions (ambient room temperature and humidity), and create a work order for the various laboratory departments to initiate testing. The lab analyses will begin within several days of the coating application.

6.0 ANALYTICAL PROCEDURES AND CALIBRATION

6.1 Facility and Laboratory Testing and Calibration

CTC has developed and currently maintains a calibration system within the factory and the laboratory. Testing and measuring equipment are calibrated on a periodic basis to ensure the accuracy of the collected data.

6.1.1 Facility Testing and Calibration

Calibration procedures within the factory are derived from ISO 10012-1 and MIL SPEC 45662A guidelines. A software package is used to track calibration information for each piece of testing and measuring equipment. This software serves to alert personnel when each piece of equipment is scheduled for calibration. Certified solutions and reference materials traceable to NIST are purchased when they are available. Where a suitable source of material does not exist, a secondary standard is prepared and a true value obtained by measurement against a NISTtraceable standard.

6.1.2 Laboratory Testing and Calibration Procedures

The analytical methods performed at *CTC* are adapted from standard ASTM, MIL-SPEC, EPA, Association of Official Analytical Chemists (AOAC), and/or industry protocols for similar manufacturing operations. Initial calibration and periodic calibration verification are performed at the frequencies specified by the methodology to ensure an instrument is operating sufficiently to meet sensitivity and selectivity requirements. At a minimum, all equipment is calibrated before use and is verified during use and/or immediately after each sample batch. Standard solutions are purchased from reputable chemical supply houses in neat, diluted forms. Where certified and traceable to NIST reference materials and solutions are available, the laboratory purchases these for calibration and standardization. Data from all equipment calibrations and chemical standard certificates from vendors are stored in laboratory files and are readily retrievable. Each calibration procedure is documented as a formal standard laboratory operating procedure for which the analyst conducting experiments is trained. The analyst is also trained to detect nonconforming calibrations from method-specific QA checks. No samples are reported in which the full calibration curve, or the periodic calibration check standards, are outside method performance standards.

6.2 Product Quality Procedures

Each apparatus that will be used to assess the quality of a coating on a test panel is set up and maintained according to each manufacturer's instructions and/or the published reference methods. Actual sample analysis will be conducted only after setup is verified per the reference method and the equipment manufacturer's instructions. As available, samples of known materials with established product qualities are used to verify that a system is functioning properly. For example, traceable thickness standards are used to calibrate the eddy current thickness instrument. Applicable ASTM methods are listed in Appendix E.

6.3 Work Instructions (Standard Operating Procedures) and Calibration

Tables 11 and 12 summarize the methods and calibration criteria that will be used to evaluate the coatings. The laboratory creates a standard operating procedure (SOP) for each test that it performs on a routine basis adapted from published references, such as ASTM and EPA, and from accepted protocols provided by industrial suppliers. SOPs are in the form of ISO 9000 Work Instructions. Work Instructions are created for equipment operation/sample analysis instructions, calibration, and maintenance. The Laboratory Manager ensures that Work Instructions are created, reviewed, and followed by laboratory personnel. The Work Instructions adhere to the quality elements contained in the original reference sources. The format for a laboratory Work Instruction is as follows:

- Title, Controlled ID #, Revision #
- Purpose
- Applicability
- Summary of Method
- Definitions
- Supporting Documents

- Equipment and Materials
- Training
- Environment, Health and Safety
- Calibration and Verification
- Maintenance
- Instruction/Process.

Non-sitiant Frates	Matha J	Matha J	Calibration	Calibratian	Calibratian
Noncritical Factor	Method	Type	Procedure	Erequency	Accept Criteria ⁽¹⁾
Input Air Pressure	Factory gauge	Pressure gauge	Comparison to NIST-traceable standard	Six months	±5 psig
Products Involved in Testing	Standard Test Panels	N/A	N/A	N/A	N/A
Zinc Phosphate Pretreatment Weight	ASTM B 767	Chromate solution 50g/L CrO ₃	Comparison to NIST-traceable standard	With each use	80-120%
Surface Area of Each Panel	Ruler	Ruler	Inspect for damage, replace if necessary	With each use	N/A
Ambient Factory Relative Humidity	Thermal Hygrometer	Thermal Hygrometer	Sent for calibration or certification	Annually	N/A
Ambient Factory Temperature	Thermal Hygrometer	Thermal Hygrometer	Sent for calibration or certification	Annually	N/A
Spray Booth Relative Humidity	Thermal Hygrometer	Thermal Hygrometer	Sent for calibration or certification	Annually	N/A
Spray Booth Temperature	Thermal Hygrometer	Thermal Hygrometer	Sent for calibration or certification	Annually	N/A
Spray Booth Air Velocity	per ACGIH	Anemometer	Sent for calibration or certification	Annually	N/A
Distance to Panels	Ruler	Ruler	Inspect for damage, replace if necessary	With each use	N/A
Temperature of Panels, as Coated	IR Thermometer	IR Thermometer	Sent for calibration or certification	Annually	N/A
Horizontal Gun Traverse Speed	Stopwatch	Stopwatch	Sent for calibration or certification	Six months	N/A
Vertical Drop Between Passes	Ruler	Ruler	Inspect for damage, replace if necessary	With each use	N/A
Dwell Time Between Passes	Stopwatch	Stopwatch	Sent for calibration or certification	Six months	N/A
VOC Content of Applied Coating	ASTM D 3960	Volatile content	Comparison to NIST-traceable standard	With each use	±0.003 g
Density of Applied Coating	ASTM D 1475	Weight	Comparison to NIST-traceable standard	With each use	±0.003 g
% Solids of Applied Coating	ASTM D 2369	Weight	Comparison to NIST-traceable standard	With each use	±0.003 g
Coating Temperature, as Applied	Thermometer	Thermometer	Comparison to NIST-traceable standard	Annually	±0.2 °C
Coating Viscosity, as Applied	ASTM D 1200	Ford Cup	Comparison to NIST-traceable standard	Prior to each test	±10%
Oven Temperature	Thermocouple	Thermocouple/ (Controllers)	Comparison to NIST-traceable standard	Annually/ (Six months)	±2.2 °C/ (±0.8 °C)
Oven Cure Time	Stopwatch	Stopwatch	Sent for calibration or certification	Six months	N/A

Table 11. Noncritical Control Factor Testing and Calibration Criteria

(1) As a percent recovery of a standard. N/A = Not Applicable

Critical Measurement	Method Number ^a	Method Type	Calibration Procedure	Calibration Frequency	Calibration Accept. Criteria ^b
Test Cap Air Pressure ^c	Manufacturer's recommendation	Test Cap	Manufacturer's recommendation	Manufacturer's recommendation	Manufacturer's recommendation ^d
Paint Flow Rate ^e	Manufacturer's recommendation	Flow Meter	Comparison to NIST-traceable standard	Six months	N/A
Total Paint Flow ^e	Manufacturer's recommendation	Flow Meter	Comparison to NIST-traceable standard	Six months	N/A
Dry film Thickness (DFT)	ASTM B 499	Magnetic	Comparison to NIST-traceable standard	Verify calibration after each run	90-110%
DFT Variation	N/A	N/A	N/A	N/A	N/A
Gloss	ASTM D 523	Glossmeter	Comparison to NIST-traceable standard	Verify calibration after each run	90-110%
Distinctness of Image (DOI) ^f	ASTM D 5767 Method B ^g	Image analyzer	Manufacturer's recommendation	Manufacturer's recommendation	Manufacturer's recommendation
Visual Appearance	N/A	Visual	N/A	N/A	N/A
Transfer Efficiency (TE)	ASTM D 5286 Test Method A	Weight	Comparison to NIST-traceable standard	Verify calibration prior to each use	±3.0 g

Table 12. Critical Response Factor Testing and Calibration Criteria

a Listing of ASTM methods to be used is provided in Appendix E. b As a percent recovery of a standard.

c HVLP tests only. d HVLP vendor provides documentation of calibration device. e Pressure feed fluid delivery systems only. f Performed by ACT Laboratories, Inc.

g Except: rotating eight-bladed disc replaces sliding combed shutter. N/A = Not Applicable.

6.4 **Nonstandard Methods**

For nonstandard methods (i.e., no commonly accepted or specified method exists or no traceable calibration materials exist), procedures will be performed according to the manufacturer's instructions or to the best capabilities of the equipment and the laboratory. This information will be documented in an SOP format. The performance will be judged based on the manufacturer's specifications or in-house-developed protocols. These protocols will be similar or representative in magnitude and scope to related methods performed in the laboratory, which do have reference performance criteria for precision and accuracy. For instance, if a nonstandard quantitative chemical procedure is being performed, it should produce replicate results of +/- 25 relative percent difference and should give values within +/- 20 percent of true or expected values for calibration and percent recovery check samples. For qualitative procedures, replicate results should agree as to their final evaluations of quality or performance (i.e., both should either pass or both should fail if sampled together from a properly functioning process). The intended use and any limitations would be explained in an SOP for a nonstandard procedure. For this project, however, CTC does not intend to use any nonstandard methods.

7.0 DATA REDUCTION, VALIDATION, AND REPORTING

7.1 Raw Data Handling

Raw data will be generated and collected by the analysts at the bench and/or process level. Process data is recorded into a process log during factory operations. Bench data will include original observations, printouts, and readouts from equipment for sample, standard, and reference quality control (QC) analyses. Data will be collected both manually and electronically. At a minimum, the date, time, sample ID, instrument ID, analyst ID, raw signal or processed signal, and/or qualitative observations will be recorded. Comments documenting unusual or nonstandard observations will also be included on the forms as necessary. Raw data will be processed manually by the analyst, automatically by an electronic program, or electronically after being entered into a computer. The analyst will be responsible for scrutinizing the data according to specified precision, accuracy, and completeness policies. Raw data bench sheets, calculations, and data summary sheets will be kept together for each sample batch. From the written SOPs and raw data bench files, the steps leading to a final result may be traced.

7.2 Preliminary Data Package Validation

The generating analyst will assemble a preliminary data package. This package will contain the QC and raw data results, calculations, electronic printouts, conclusions, and laboratory sample tracking information. A second analyst will review the entire package and may also check sample and storage logs, standard logs, calibration logs, and other files, as necessary, to ensure that tracking, sample treatments, and calculations are correct. After the package has been peer reviewed in this manner, a preliminary data report will be prepared. The entire package and final report will be submitted to the Laboratory Manager.

7.3 Final Data Validation

The Laboratory Manager will ultimately be responsible for all final data released from the laboratory. The Laboratory Manager will review the final results for adequacy to project QA objectives. If the manager suspects an anomaly or nonconcurrence with expected or historical performance values, project QA objectives, or method-specific QA requirements of the laboratory SOP, he or she will initiate a second review of the raw data and query the generating and reviewing analysts about the nonconformance. The Laboratory Manager will also request specific corrective action. If suspicion about data validity still exists after internal review of laboratory records, the manager may authorize a reanalysis. If sufficient sample is not available for retesting, a resampling will occur. If the sampling window has passed, or resampling is not possible, the Laboratory Manager. The Laboratory Manager will flag the data as suspect and notify the Technical Project Manager. The Laboratory Manager will sign and date the final data package.

7.4 Data Reduction

The test panels coated during the HVLP verification test will be evaluated for both finish quality and TE. The finish quality data obtained from the HVLP panels will be compared to a coating reference panel to determine whether the HVLP spray gun was able to provide an acceptable quality finish. The finish quality of the HVLP and CAS baseline panels will be compared only to show that the finish quality of the baseline panels was not sacrificed to obtain higher TE values for the CAS guns, unless the coating reference panel does not meet the target requirements established in the TQAPP. The TE data for the HVLP and CAS baseline test will then be compared to determine the relative improvement in TE obtained using the HVLP equipment.

The relative improvement in TE will be calculated as absolute and applied. Absolute TE is defined for this test as the actual, unadjusted TE obtained from this verification test. Absolute TE includes the coating that was sprayed between panels and when the gun was traveling toward or away from the racks. Applied TE only takes into account the coating that was sprayed while the gun was positioned directly in front of a panel. The applied TE normalizes the data to a certain extent. Applied TE estimates the results that would be obtained if each rack consisted of a single panel, 81.3 cm x 30.5 cm, and that the gun begins, or stops, spraying as the vertical axis of the spray gun crosses the leading, or trailing edge, of the panel.

The results of these verification tests will be presented in the Verification Statement as environmental and marketability factors. The environmental factors will include the relative TE improvement over the CAS baseline, a calculation of emissions reduction based on the test data, a calculation of cost savings achievable based on the test results, and the dynamic output air pressure used for the HVLP spray gun during the verification test. The marketability factors will include the results of the DFT, DOI, gloss, and visual appearance analyses for the HVLP test, the test coating reference panel, and the CAS baseline, as appropriate.

7.5 Data Reporting and Archival

The data generated by the HVLP verification and CAS baseline tests will be reviewed and validated by ETV CCEP project staff, the ETV CCEP QA Officer, and Technical Peer Reviewers. This process constitutes data validation as required by the ETV CCEP QMP. After the data have been validated, a report signed and dated by the Laboratory Manager is submitted to the Technical Project Manager, the QA Officer, and other technical principals involved in the project. The Technical Project Manager will determine the appropriateness of the data and make any interpretations with respect to project QA objectives. The final laboratory report will contain the lab sample ID, date reported, date analyzed, the analyst, the SOP used for each parameter, the process or sampling point identification, the final result, and the units. The laboratory will retain the data packages for at least 10 years. The Technical Project Manager or the Program Manager will forward the results and conclusions to EPA in their regular reports, after obtaining corporate approvals.

The ETV CCEP will then prepare a Verification Report that includes a description of the tests performed, data obtained from those tests, and the calculations made from that data. The Verification Report will also be summarized as a Verification Statement. The raw data, results of data reduction, and QA analyses will be compiled into a separate Data Notebook.

The Verification Report, Verification Statement, and Data Notebook will undergo a brief preliminary review by EPA's Pilot Manager and QA Officer for format and consistency with the test's conclusions and for final data validation. They will then be reviewed by the HVLP equipment provider. The equipment provider's comments will be incorporated into the test documents. The Verification Report, Verification Statement, and Data Notebook will then be distributed for Technical Peer Review. Comments received from the Technical Peer Reviewers will be addressed by CTC and the resolutions documented in writing. A revised Verification Report, Verification Statement, and a copy of the written documentation of the comment resolutions will then be submitted to the EPA Pilot Manager, who will arrange for Technical Editor review. The Data Notebook will not be reviewed by the EPA Technical Editor, but will instead be archived by CTC for future retrieval on public or EPA request. Approval by EPA management will be coordinated by the EPA ETV CCEP Pilot Manager and the EPA ETV Program Manager. CTC will prepare the approved Verification Report and Verification Statement for posting on the ETV website. CTC will be responsible for publishing each Verification Report and Statement.

7.6 Verification Statement

After the EPA reviews the results and conclusions from the Technical Project Manager, the Verification Statement/Verification Report will be written by *CTC*, sent to the vendor for comment, passed through technical peer review, and submitted to EPA for approval. Following agreement by the technology provider, *CTC* will disseminate the Verification Statement, which is a summary of the test results included in the Verification Report.

8.0 INTERNAL QUALITY CONTROL CHECKS

8.1 Guide Used for Internal Quality Program

CTC has established an ISO 9001 operating program for its laboratories and the Demonstration Factory. The laboratory is currently establishing a formal quality control program for its specific operations. The format for laboratory QA/QC is being adapted from several sources, as listed in Table 13.

Document	Reference Source
General Requirements for the Competence of	ISO Guide 25, ISO Quality Programs
Calibration and Testing Laboratories	
Critical Elements for Laboratories	Pennsylvania Department of
	Environmental Protection
Chapter One, Quality Control	SW-846, EPA Test Methods
Requirements of 100-300 series of methods	EPA Test Methods
Handbook of Quality Assurance for the	James P. Dux
Analytical Chemistry Laboratory, 2 nd Ed.	

Table 13. CTC Laboratory QA/QC Format Sources

8.2 Types of QA Checks

The ETF laboratory at *CTC* follows published methodologies, wherever possible, for testing protocols. Laboratory methods are adapted from Federal Specifications, Military Specifications, ASTM Test Methods, and supplier instructions. The ETF laboratory adheres to the QA/QC requirements specified in these documents. In addition, where QA/QC criteria are not specified, or where the laboratory performs additional QA/QC activities, these protocols are explained in the laboratory's SOPs (Work Instructions). Each *CTC* facility that uses supplied products implements its own level of QA/QC. *CTC*'s laboratory at ETF will perform the testing and QA/QC verification outlined in Tables 8 and 9 (Precision, Accuracy, and Completeness) and Tables 11 and 12 (Calibration); therefore, these tables should be referred to for the method-specific QA/QC that will be performed.

8.3 Basic QA Checks

During each test, an internal Process QA Checklist will be completed by the laboratory staff to ensure the appropriate parts, panels, samples, and operating conditions are used. The laboratory also monitors its reagent deionized water to ensure it meets purity levels consistent with analytical methodologies. The filters are replaced quarterly before failures are encountered. Samples are not processed until the filters are replaced when failures do occur. The quality of the water is assessed with method reagent water blanks. Blank levels must not exceed minimum detection levels for a given parameter to be considered valid for use.

Thermometers are checked against NIST-certified thermometers at two temperatures. The laboratory checks and records the temperatures of sample storage areas, ovens, hot plate operations, and certain liquid baths that use thermometers.

Balances are calibrated by an outside organization using standards traceable to NIST. *CTC* also performs in-house, periodic verifications with ASTM Class 1 weights. The ETF laboratory maintains records of the verification activities and calibration certificates. The laboratory analyst also checks the balances prior to use with ASTM Class 1 weights.

Reagents purchased directly by the laboratory are American Chemical Society (ACS) grade or better. Reagents are not used beyond their certified expiration dates. Reagents are dated on receipt and when first opened.

Laboratory waste is segregated according to chemical classifications in labeled containers to avoid cross-contamination of samples.

8.4 Specific Checks

CTC's ETF laboratory will analyze uncoated panels for dry film thickness to verify that the instrument has not drifted from zero, perform duplicate analyses on the same samples, and perform calibration checks of the laboratory equipment. Laboratory personnel will also check any referenced materials and equipment as available and specified by the referenced methodology and/or the project-specific QA/QC objectives. Laboratory records are maintained with the sample data packages and/or in centralized files, as appropriate. To ensure comparability, the laboratory will carefully control process conditions and perform product evaluation tests consistently for each specimen. The specific QA checks listed in Tables 8, 9, 11, and 12 provide the necessary data to determine whether process control and product testing objectives are being met. ASTM, Federal, and Military methods that are accepted in industry for product evaluations and supplier-endorsed methods for process control, will be used for all critical measurements, thus satisfying the QA objective. A listing of the published methods that will be used for this protocol is included in Appendix E.

9.0 PERFORMANCE AND SYSTEM AUDITS

CTC has developed a system of internal and external audits to monitor both program and project performance. These include monthly managers meetings and reports, financial statements, EPA reviews and stakeholders meetings, and In-Process Reviews. The ETF laboratory also analyzes performance evaluation samples in order to maintain Pennsylvania Department of Environmental Protection Certification.

ISO Internal Audits

CTC has established its quality system based on ISO 9000 and 14000 and has implemented a system of ISO internal audits. This information will be used for internal purposes.

On-Site Visits

The EPA Project Officer may visit *CTC* for an on-site visit during the execution of this project. All project, process, quality assurance, and laboratory testing information will be available for review.

EPA Audits

The EPA will periodically audit *CTC* during this project. All project, process, quality assurance, and laboratory testing information will be made available per the EPA's auditing procedures.

Technical Systems Audits

A listing of all coating equipment, laboratory measuring and testing devices, and procedures, coating procedures, and a copy of the approved ETV QMP and the approved ETV CCEP QMP will be given to the project QA Officer. The QA Officer will conduct an initial audit, and additional audits thereafter according to the ETV CCEP QMP, of demonstration and testing activities. The results of this activity will be forwarded to EPA in reports from the Program Manager or the Technical Project Manager.

Audits of Data Quality

Peer review in the laboratory constitutes a process whereby two analysts review raw data generated at the bench level. After data are reduced, they undergo review by laboratory management. For this protocol, laboratory management will spot check 10 percent of the project data by performing a total review from raw to final results. This activity will occur in addition to the routine management review of all data. Records will be kept to show which data have been reviewed in this manner.

10.0 CALCULATION OF DATA QUALITY INDICATORS

10.1 Precision

Duplicates will be performed on separate samples, as well as on the same sample source, depending on the method being employed. In addition, the final result for a given test may be the arithmetic mean of several determinations on the part or matrix. In this case, duplicate precision calculations will be performed on the means. The following calculations will be used to assess the precision between duplicate measurements.

Relative Percent Difference (RPD) = $[(C1 - C2) \times 100\%] / [(C1 + C2) / 2]$

where: C1 = larger of the two observations C2 = smaller of the two observations

Relative Standard Deviation (RSD) = $(s/y) \times 100\%$

where: s = standard deviationy = mean of replicates.

10.2 Accuracy

Accuracy will be determined as percent recovery of a check standard, check sample, or matrix spike.

For matrix spikes and synthetic check samples:

Percent Recovery (%R) = 100% x [(S - U)/T]

where: S = observed concentration in spiked sample U = observed concentration in unspiked sample T = true value of spike added to sample.

For standard reference materials (srm) used as calibration checks:

% $R = 100\% x (C_m / C_{srm})$

where: C_m = observed concentration of reference material C_{srm} = theoretical value of srm.

10.3 Completeness

Percent Completeness (%C) = 100% x (V/T)

where: V = number of determinations judged valid T = total number of determinations for a given method type.

10.4 Project Specific Indicators

Process control limit: range specified by supplier for a given process parameter.

11.0 CORRECTIVE ACTION

11.1 Routine Corrective Action

Routine corrective action will be undertaken in the event that a parameter in Tables 8, 9, 11, and 12 is outside the prescribed limits specified in these tables, or when a process parameter is beyond specified control limits. Examples of nonconformances include, but are not limited to, invalid calibration data, inadvertent failure to perform method-specific QA tests, process control data outside specified control limits, and failed precision and/or accuracy indicators. Such nonconformances will be documented on a standard laboratory form. Corrective action will involve taking all necessary steps to restore a measuring system to proper working order and summarizing the corrective action and results of subsequent system verifications on a standard form. Some nonconformances will be detected while analysis or sample processing is in progress, and can be rectified in real time at the bench level. Other nonconformances may be detected only after a processing trial and/or sample analyses are completed. These types of nonconformances are typically detected at the Laboratory Manager level of data review. In all cases of nonconformance, the Laboratory Manager will consider repeating the sample analysis as one method of corrective action. If a sufficient sample is not available, or the holding time has been exceeded, complete reprocessing may be ordered to generate new samples if a determination is made by the Technical Project Manager that the nonconformance jeopardizes the integrity of the conclusions to be drawn from the data. In all cases, a nonconformance will be rectified before sample processing and analysis continues. If corrective action does not restore the production or analytical system, causing a deviation from the ETV CCEP QMP, CTC will contact the EPA Project Contract Officer. In cases of routine nonconformance, EPA will be notified in the Program Manager's or Technical Project Manager's regular report to the EPA Project Contract Officer. A complete discussion will accompany each nonconformance.

11.2 Nonroutine Corrective Action

While not anticipated, activities such as internal audits by the facility QA Officer, and on-site visits by the EPA Project Contract Officer, may result in findings that contradict deliverables in the ETV CCEP QMP. In the event that nonconformances are detected by bodies outside the laboratory organizational unit, as for routine nonconformances, these problems will be rectified and documented prior to processing or analyzing further samples or specimens.

12.0 QUALITY CONTROL REPORTS TO MANAGEMENT

As shown on the Project Organization Chart in Figure 5, *CTC* employs a full-time QA Officer who is independent from the project management team. It is the responsibility of the QA Officer to monitor *CTC* Demonstration Projects for adherence to project specific QMPs. The Laboratory Manager monitors the operation of the laboratory on a daily basis and provides comments to the QA Officer to facilitate his activities. The QA Officer will audit the operation records, laboratory records, and laboratory data reports and provide a written report of the findings to the Technical Project Manager and Laboratory Manager. The Technical Project Manager will ensure these reports are included in the report to the EPA. The Laboratory Manager will be responsible for achieving closure on items addressed in the report. Specific items to be addressed and discussed in the QA report include the following:

- General assessment of data quality in terms of general QA objectives in Section 4.1
- Specific assessment of data quality in terms of quantitative and qualitative indicators listed in Sections 4.2 and 4.3
- Listing and summary of all nonconformances and/or deviations from the ETV CCEP QMP
- Impact of nonconformances on data quality
- Listing and summary of corrective actions
- Results of internal QA audits
- Closure of open items from last report or communications with EPA in current reporting period
- Deviations or changes in the ETV CCEP QMP
- Progress of CTC QA Programs in relation to current project
- Limitations on conclusions, use of the data
- Planned QA activities, open items for next reporting period.

13.0 REFERENCES

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APPENDIX A

Apparatus Setup

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APPENDIX B

Equipment Testing Location

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APPENDIX C

Standard Test Panel

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APPENDIX D

Coating Specifications

Coating Specifications for ETV CCEP HVLP Equipment Verification Testing

Default Standard Test Panels:

Substrate:	Cold-rolled steel SAE 1008 specification Received unpolished and untreated
Pretreatment:	Zinc Phosphate
Application:	HVLP and Conventional Air Spray
VOC Content:	Must meet the regulatory requirements of the target industry Must not contain water or exempt solvents
Cure Method:	Recommended by coating manufacturer
Target DFT:	Recommended by coating manufacturer
Notes:	Coating must not contain lead or chromate.

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APPENDIX E

ASTM Methods
ASTM Methods

ASTM B 499	Standard Test Method for Measurement of Coating Thicknesses by the Magnetic Method: Nonmagnetic Coatings on Magnetic Basis Metals
ASTM D 523	Standard Test Method for Specular Gloss
ASTM B 767	Standard Guide for Determining Mass per Unit Area of Electrodeposited and Related Coatings by Gravimetric and other Chemical Analysis Procedures
ASTM D 1200	Standard Test Method for Viscosity by Ford Viscosity Cup
ASTM D 1475	Standard Test Method for Density of Paint, Varnish, Lacquer, and Related Products
ASTM D 2369	Standard Test Method for Volatile Content of Coatings
ASTM D 3960	Standard Practice for Determining Volatile Organic Compound (VOC) Content of Paints and Related Coatings
ASTM D 5286	Standard Test Methods for Determination of Transfer Efficiency Under General Production Conditions for Spray Application of Paints
ASTM D 5767	Standard Test Methods for Instrumental Measurement of Distinctness-of- Image Gloss of Coating Surfaces

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APPENDIX F

CTC Work Instructions

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- -- Job Safety Analysis for Grit Blast Booth
- 0926.005 -- Pre-treatment System Operation (equipment)
- 0926.001 -- Pretreatment Line Operation (testing)
- 0926.018 -- Wet Spray Booth Operation
- 0931.017 -- Cold-Rolled Steel Coating Weights
- 0931.012 -- Viscosity
- 0931.001 -- Density
- 0931.013 -- Determination of Weight Percent Solids of Paint
- 0931.014 -- Determination of Transfer Efficiency for the Spray Application of Paint
- 0931.019 -- Gloss of Organic Coatings
- 0931.020 -- Measurement of Coating Thickness by the Magnetic Method